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FABRICATION OF NOVEL COMPOSITES - PART II: FABRICATION AND PROPERTIES OF Ba-MICA/Al$_2$O$_3$ COMPOSITES

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CERAMICS RESEARCH DIVISION

May 1973

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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Technical Report by
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FABRICATION OF NOVEL COMPOSITES. PART II: FABRICATION AND PROPERTIES OF Ba-MICA/Al_2O_3 COMPOSITES

ABSTRACT

The feasibility of fabricating lamellar ceramic-based composites with controllable and easily modified properties by reactive hot pressing of gamma-Al_2O_3 with Ba-mica ($BaMg_3Al_2Si_2O_{10}F_2$) has been successfully demonstrated. Cylindrical disks 2 inches in diameter by 1/4-inch thick of Ba-mica/Al_2O_3 composite material with greater than 99.5% of theoretical density have been produced with the following range of properties: $Knoop_{100}$ hardness from 950 to 1970 kg/mm^2, four-point flexure strength from 38,000 to 53,500 psi, and Young's modulus from 28.6 to 60.5 × 10^6 psi.

CONTENTS

I. INTRODUCTION

Design engineers have been reluctant to utilize ceramic materials for many potential applications because of their brittle characteristics and inflexibility of properties. Materials are screened according to certain minimum property requirements based on the particular application, such as armor, radomes, bearings (high and low temperature), and turbine engine components. In general, ceramic scientists fabricate new materials with rigid properties and oftentimes unknown applications. In this paper a new but proven concept of material fabrication is described whereby various properties can be easily adjusted which will permit designers new degrees of freedom in meeting complex engineering requirements. By this technique materials can be tailor-made for the particular application.

Most ceramic materials, like alumina (Al_2O_3) and magnesia (MgO), exhibit many desirable properties, such as light weight, high temperature stability, oxidation resistance, and high strength and stiffness, but they are extremely brittle and difficult to machine. In order to moderate their brittle characteristics, various investigators have been studying the possibility of incorporating ductile or flexible components into ceramics.[1-3] It has been demonstrated that these composites exhibit modified brittle properties; they yield plastically before catastrophic failure. Other workers have been analyzing the beneficial effect of a dispersed second phase (including cracks or voids) on the toughening[4,5] and thermal shock resistance[6,7] of ceramics. All of the preceding studies clearly show that the properties of ceramics can be substantially improved, controlled, and predicted by the systematic incorporation of a second phase.

II. BRIEF HISTORY OF MICA RESEARCH

A unique family of materials which have been widely overlooked as candidates for second phase incorporation into ceramics are synthetic fluorine micas,[8] high temperature dielectric materials which exhibit flexibility in thin sheets. Micas, like graphite and MoS_2, are also natural solid lubricants because of their platy structure and easy cleavage. In the past, single crystals of natural mica (isinglass) and mica ceramics have been utilized widely for electronic and thermal insulation applications. The addition of carefully selected micas to certain high strength ceramics should modify the brittle characteristics while retaining most of the strength properties and enhancing the machinability. Further, if the micas can be incorporated without any significant formation of reaction products at the matrix-mica interface, the final composite system is directly analogous to a ceramic with a dispersion of cracks; the cracks in this case, however, are totally filled with mica plates, potentially enhancing the thermal shock resistance, while retaining the strength. Finally, incorporation of mica into ceramics (or even metals and polymers) should improve their wear resistance because of their natural self-lubricating properties.

The Russians[9,10] and Japanese[11,12] have already begun to investigate the potential of utilizing micas as components in composites. One study[10] reported on the successful use of mica/silver composites as solid lubricants in hard vacuum, where the requirements consisted of no magnetic susceptibility and

good heat and electrical conduction. Another effort[9] demonstrated an order of magnitude improvement in wear resistance of mica/BN composites over standard materials; this result suggests that mica/BN can be used in the sealing points of high-temperature compressors. The United States Government has sponsored large research and development programs on mica, primarily emphasizing the production of large single crystals.[13-15] Extensive programs have also been carried out in Japan and Russia, but none of these efforts have lead to commercial production of large single crystals.[16] The United States Bureau of Mines, however, did demonstrate that machinable mica ceramics could be fabricated, as well as mica/Al_2O_3, mica/BN, mica/Ni, and mica/fluoramphibole composites.[13] The composite work was performed at the end of the program and was very limited; little characterization was carried out on these materials. Their research, however, did demonstrate that over a hundred micas with varying chemical compositions and properties could be synthesized in powder form.

The chemical formula for mica is $X_2Y_{4-6}Z_8O_{20}$ $(OH,F,Cl)_4$,

where

$$X = Na^+, K^+, Rb^+, Cs^+, Ca^+, Sr^{2+}, Pb^{2+}, (La^{3+}), etc.,$$

$$Y = Al^{3+}, Fe^{3+}, Fe^{2+}, Mg^{2+}, Ni^{2+}, Li^+, etc., and$$

$$Z = Si^{4+}, Ge^{4+}, Al^{3+}, Fe^{3+}, B^{3+}, Be^{2+}, etc.$$

Large modifications in the properties of mica can be produced by appropriately varying the mica chemical composition. For example, the substitution of Ba for K in mica causes a change in melting point of 100 C and a change in the $Knoop_{100}$ microhardness of 300 Kg/mm^2. Substitution of fluorine for hydroxyl (common in natural micas) doubles the thermal stability. This variability in mica composition as well as properties also implies that composite formation with mica can be optimized by the appropriate choice of mica. Further, it is also conceivable that the properties of a mica composite can be adjusted by varying the volume as well as the composition of the incorporated mica. Data from the Bureau of Mines[13] clearly illustrates this point: a $KMg_3AlSi_3O_{10}F_2$/Ni composite had a bend strength of 11,700 psi, whereas a $BaMg_3Al_2Si_2O_{10}F_2$/Ni composite had a strength of 31,600 psi.

A basic research program was initiated at the Army Materials and Mechanics Research Center in 1966 to establish enough expertise so that mica composites could be successfully fabricated. The solution of the crystal structures of $KMg_3AlSi_3O_{10}F_2$ and $BaLiMg_2AlSi_3O_{10}F_2$ by McCauley[17] lead to the derivation of mathematical formulae which can be used to predict the crystal structure and some of the properties of any mica.[18] Further, the relation of the properties of micas to their crystal structures was more fully elucidated by these crystallographic investigations.[19]

III. HOT PRESSING OF $KMg_3AlSi_3O_{10}F_2$ WITH $\gamma-Al_2O_3$

Alumina (Al_2O_3) is a widely used ceramic material, but is restricted i some of its potential applications because of its relatively low thermal sh(resistance and impact strength. Addition of mica to this material could imp these properties, as well as its friction behavior. Hence, work was initiat(on the hot pressing of Al_2O_3 with synthetic fluorine micas.[20,21] Fluorophlog pite ($KMg_3AlSi_3O_{10}F_2$)* subsequently referred to as K-mica, was chosen as the initial mica to be studied because of its ready availability.

Research performed at AMMRC by Gazza, et al.[22] on the fabrication of close to fully dense $\alpha-Al_2O_3$ ceramics using "gamma"-Al_2O_3† motivated the use of this material in the present investigation. X-ray diffraction analysis of this gamma material revealed it to be an almost amorphous material with very broad X-ray peaks suggesting that boehmite (AlO(OH)) was the predominant crystal form. The average grain size of the material is about 0.5 µm.

The success that the Bureau of Mines achieved with hot pressing of mica ceramics stimulated the use of this standard technique in this program. Mixtures of Al_2O_3 and K-mica were blended by tumbling the dry material in a glass container for at least 24 hours. The two components were mixed together in various volume percentages to facilitate rule-of-mixtures calculations. After blending, the mixtures were loaded into a graphite die and brought to the final pressure immediately. When the mixture was under full pressure, the graphite die and plunger assembly was inductively heated to the desired temperature. After 60 minutes at this temperature and pressure the power was shut off; when the temperature of the system reached about 500 C the pressure was fully released.

Figure 1 illustrates the densities measured on various 1-inch diameter by about 1/8-inch thick cylindrical disks of K-mica/Al_2O_3 composites fabricated by the preceding procedure. The bottom curve in the figure represents runs carried out at 1000 psi and 1250 C, while the top three curves, showing significantly improved densities, signify data obtained on compacts hot pressed at 7000 psi and 1175, 1200, and 1225 C. Initial temperature and pressure conditions were deduced from previous hot-pressing experiments at the Bureau of Mines.[8-13] Ideal densities (i.e., no porosity) for various K-mica/Al_2O_3 mixtures calculated by using the rule-of-mixtures formula are also plotted on this figure for comparison purposes. The following rule-of-mixtures formula assumes a linear combination and neglects any synergistic effects as well as the formation of reaction products:

$$P_c = P_1V_1 + P_2V_2$$

where

$$P_c = \text{ideal density of composite,}$$

*Obtained from Mykroy Ceramics, Ledgewood, New Jersey
†Obtained from the Republic Foil Corp., Danbury, Connecticut

P_i = density of end members, and

V_i = volume fractions of end members in mixture.

Other properties such as hardness and stiffness (Young's modulus) can also be calculated using this relationship. Table I lists the property data of Al_2O_3 and K-mica used for these calculations; property data are also tabulated for a Ba-mica and $MgAl_2O_4$ (spinel) to be referred to later in the paper.

Since the "gamma" alumina contained variable amounts of adsorbed and chemical water, the K-mica/Al_2O_3 mixtures are nominal but are accurate to within about 5%. The K-mica used for these experiments averaged about 200 μm in diameter by about 20 μm in thickness. Work is now underway using mica powder of approximately 50 to 75 μm in diameter.

In Figure 1, the cluster of points at 60 volume percent Al_2O_3 represents the formation of a glassy phase, found by electron probe analysis to be a $K_2O-Al_2O_3-SiO_2$ glass. The density data illustrates the quite marked effect that mica has on this system, especially those runs carried out at 7000 psi.
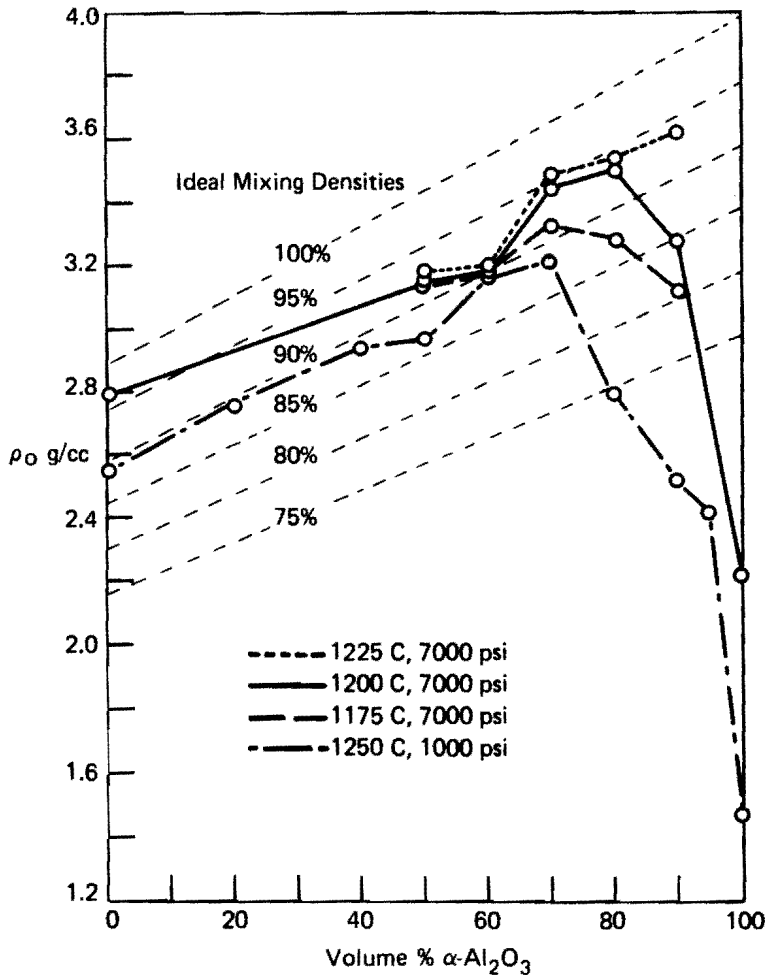

Figure 1. Observed densities of K-Mica/Al_2O_3 composites

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Table I. SELECTED PROPERTIES OF K AND Ba MICAS, αAl_2O_3 AND $MgAl_2O_4$.

	$KMg_3AlSi_3O_{10}F_2$	$BaMg_3Al_2Si_2O_{10}F_2$	α-Al_2O_3	$MgAl_2O_4$
ρ	2.80 g/cm^3	3.50 g/cm^3	3.98 g/cm^3	3.59 g/cm^3
Melting Point	1388°C	1461°C	2050°C	2105°C
Flexural Strength	10x10^3 psi	19x10^3 psi	~65x10^3 psi	—
Tensile Strength	7.5x10^3 psi	—	~40x10^3 psi	~19x10^3 psi
Young's Modulus	7.1x10^6 psi	(~15x10^6 psi?)	~55x10^6 psi	~40x10^6 psi
Knoop (100) Hardness	50	340	2000	1200
Thermal Expansion (x 10^6 in/in°F) 70-1000 F	~5.6	4.5-5.7	4.4	5.0
1000-1800 F	~5.6	~5.7	5.0	5.0
1800-3200 F	—	~5.8	6.6	—

These effects are also reflected by mean Knoop$_{100}$ microhardness values plotted in Figure 2. Standard errors are not illustrated because they would clutter the figure; they averaged about 200 kg/mm^2. Each point represents from 15 to 20 randomized measurements. The final phase assemblage, excluding glass, determined by X-ray diffraction is also noted on the figure; spinel ($MgAl_2O_4$) is the primary reaction product. Runs at 1225 C or above results in the reaction of almost all the mica to spinel and the loss of 80 weight percent of fluorine. Reaction between the K-mica and Al_2O_3 is further evidenced in Figure 3 which illustrates the change in density of 50-50 K-mica/Al_2O_3 compacts hot pressed at 1000 psi and various temperatures. Density is seen to be a linear function of temperature up to 1150 C, but noticeably changes when spinel appears as a reaction product in the run at 1250 C.

Figure 4a is a photomicrograph of a relief polished cross section of a K-mica/Al_2O_3 compact with 90 volume percent of Al_2O_3 hot pressed at 1175 C and 7000 psi. The mica flakes are all aligned nearly parallel to the top of the disk and are all fairly smooth, indicating little reaction to spinel. Figure 4b shows an identical mixture fabricated at 1225 C and 7000 psi; note the top surface of the disk at the top of the photograph. Electron probe analysis of this sample revealed that the lenticular masses are spinel, while the matrix is Al_2O_3. An unbalanced reaction which could result in this assemblage is tentatively worked out below.

$$Al_2O_3 + KMg_3AlSi_3O_{10}F_2 \rightarrow Al_2O_3 + MgAl_2O_4 + SiF_4 \uparrow + KF \uparrow + glass.$$
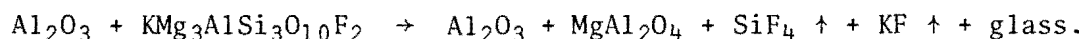
Both SiF_4 and KF vaporize and enhance sintering of the compact. A negligible amount of glass is also formed, which results in a liquid phase sintering; the glass could not be absolutely located in the sample, but seems to be located between the spinel and the Al_2O_3. Figures 1 and 2 show that the density and hardness
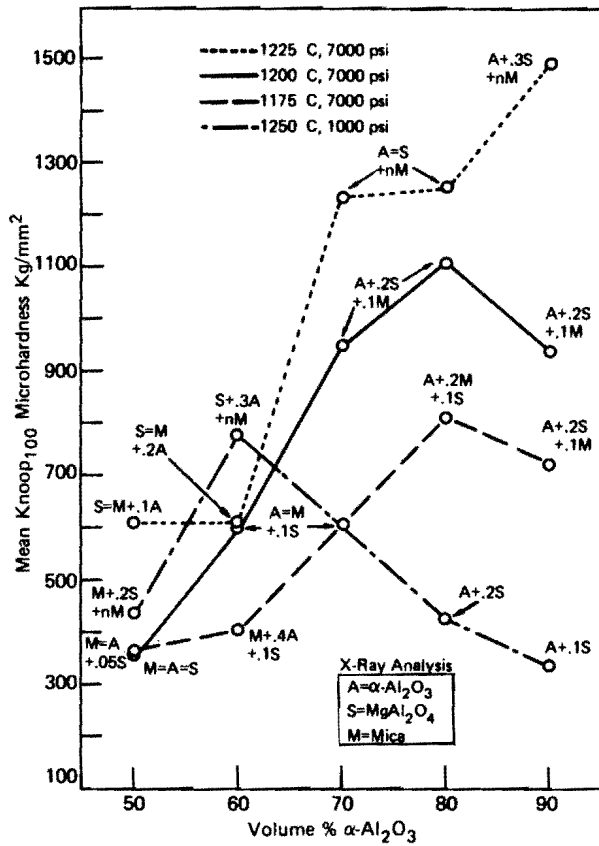
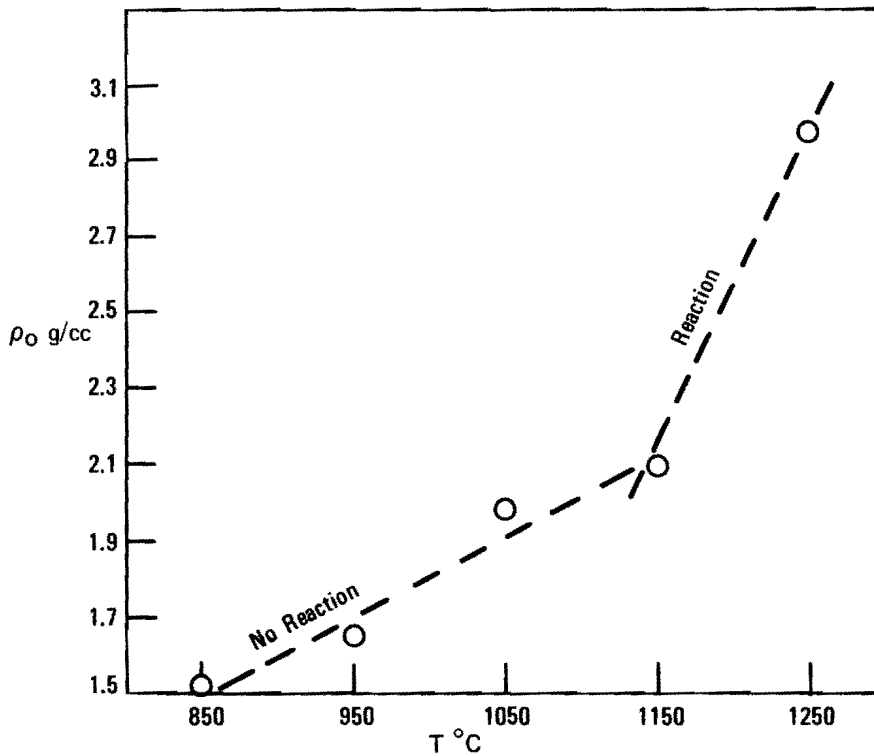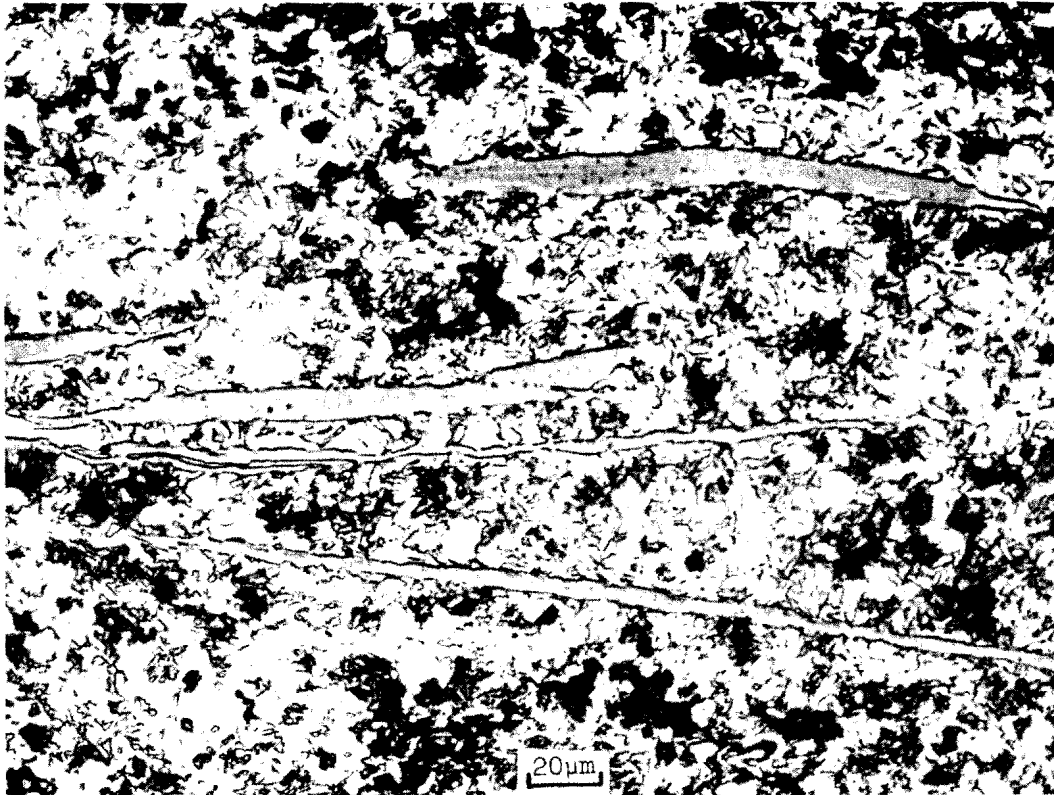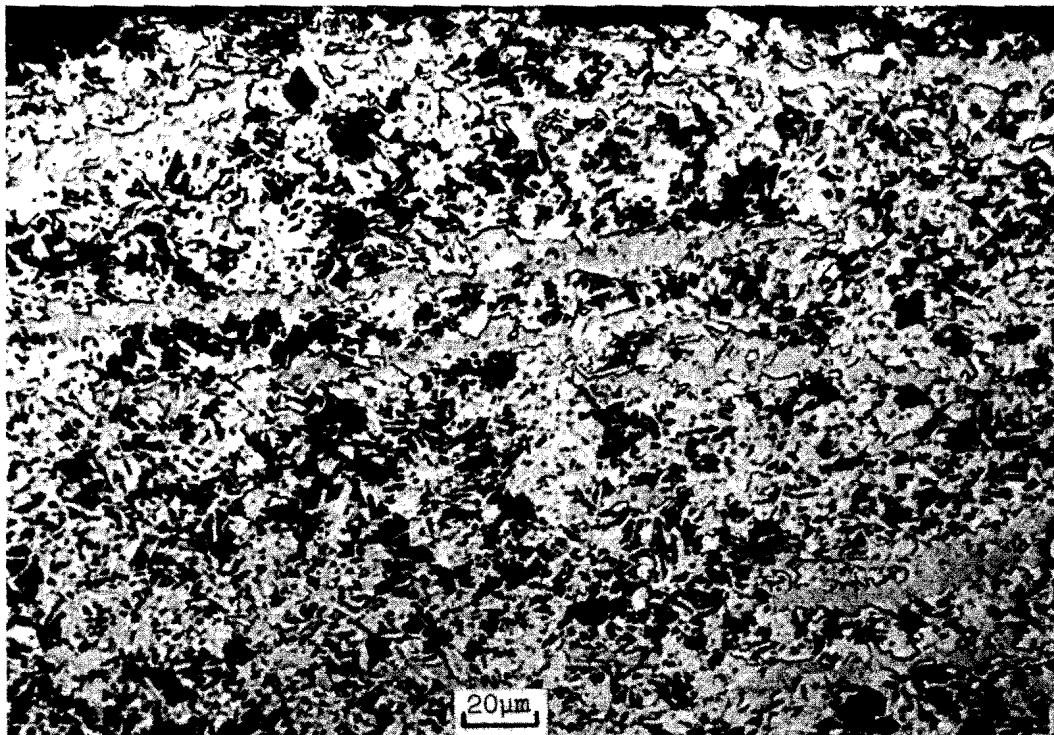Figure 2. Mean $Knoop_{100}$ microhardness of K-Mica/Al_2O_3 composites

Figure 3. Observed densities of 50-50 K-Mica/Al_2O_3 composites hot pressed at 1000 psi and various temperatures

a. Hot pressed at
1175 C and
7000 psi

b. Hot pressed at
1225 C and
7000 psi

Figure 4. Photomicrographs of K-Mica/Al$_2$O$_3$ compacts with 90 vol% Al$_2$O$_3$
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of the 90 volume percent Al_2O_3 composite hot pressed at 1225 C and 7000 psi fall between the listed values of spinel and alumina. Hence, the possibility arises that this process allows for the fabrication of eutectic-like $MgAl_2O_4/Al_2O_3$ composites about 700 C below the eutectic temperature.

The preceding results indicate that it is not possible to fabricate fully dense K-mica/Al_2O_3 composites, although the investigation did reveal a significant processing improvement for $MgAl_2O_4/Al_2O_3$ composites. In order to retard the formation of spinel, a higher melting mica, $BaMg_3Al_2Si_2O_{10}F_2$, was chosen for the next phase of the study. This will be referred to as Ba-mica and its properties are listed in Table I.

IV. HOT PRESSING OF $BaMg_3Al_2Si_2O_{10}F_2$ WITH γ-Al_2O_3

The same processing procedures which were utilized in Section III were also applied in this part of the investigation. Ba-mica* and γ-Al_2O_3,† with an average grain size of 0.2 μm, have been employed as starting materials. The X-ray diffraction powder pattern of this material exhibited fairly sharp peaks of an almost true γ-Al_2O_3 phase.

After successful hot pressing of 1-inch-diameter Ba-mica/Al_2O_3 disks, scale-up to 2" disks was also routinely accomplished. Figure 5 illustrates a set of these disks hot pressed at 1250 C and 7000 psi; the dark rings on the disks are graphite plunger marks and can be polished off if necessary. Hot pressing at temperatures above 1250 C results in the formation of spinel. This is illustrated in Figure 6, which shows a compact hot pressed at 1250 C and one at 1300 C. Note the large euhedral grains of spinel in Figure 6b and the presence of polishing pull-out (the dark areas) which was also seen in the photomicrographs of K-mica/Al_2O_3 compacts. The photomicrograph in Figure 6a shows well aligned and unreacted Ba-mica in an Al_2O_3 matrix. X-ray diffraction analysis confirmed these conclusions.
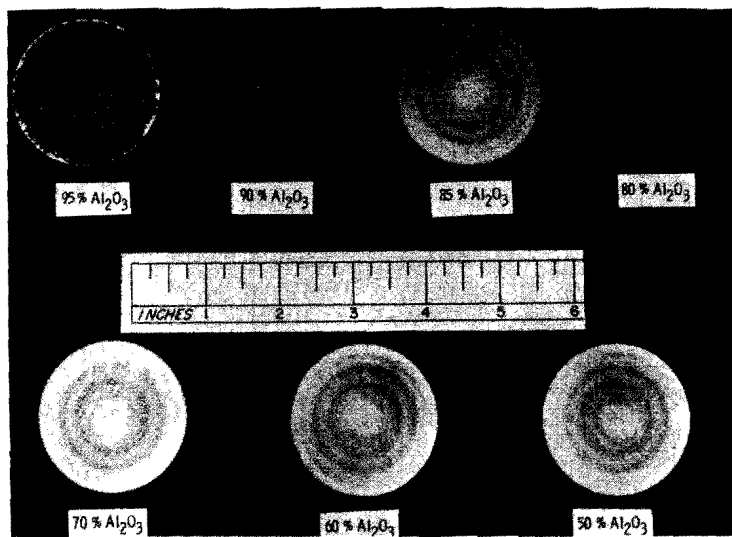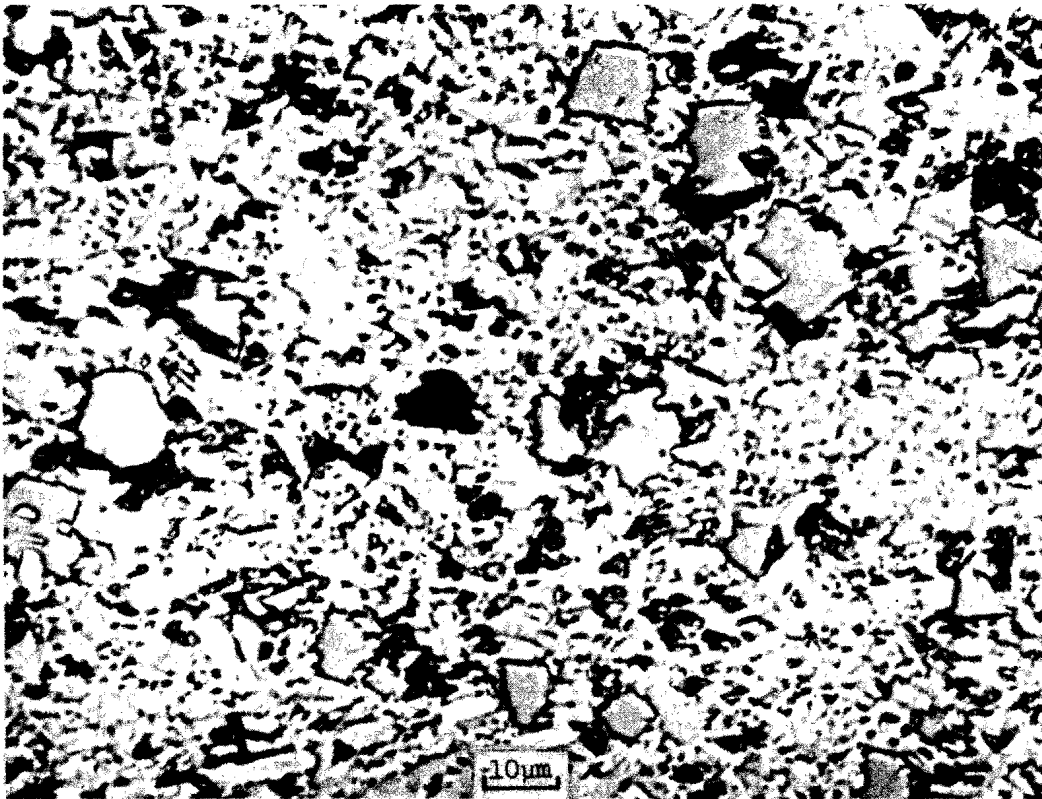

Figure 5. Disks (about ¼ inch thick) of Ba-Mica/Al_2O_3 composites hot pressed at 1250 C and 7000 psi
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*Obtained from Mykroy Ceramics, Ledgewood, New Jersey
†Obtained from Cerac Co., Menomonee Falls, Wisconsin

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a. Hot pressed at
1250 C and
7000 psi

b. Hot pressed at
1300 C and
7000 psi

Figure 6. Photomicrographs of Ba-Mica/Al$_2$O$_3$ compacts with 30 vol % 30 μm Ba-Mica
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Figure 7 is a photomicrograph of a 60 volume percent Al_2O_3 compact illustrating the homogeneous distribution and alignment of the 30 μm Ba-mica flakes. Photomicrographs in Figure 8 depict the microstructure of a 95% Al_2O_3 compact; Figure 8a was prepared by multiple relief polishing, whereas 8b was etched in boiling phosphoric acid to bring out the duplex grain size microstructure of the Al_2O_3 matrix.

Figure 9 depicts densities measured on Ba-mica/Al_2O_3 composites fabricated at 1250 C and 7000 psi. Note the close proximity of the composites to ideal mixing densities and the linear relationship between the various mixtures, indicating a close adherence to the rule of mixtures. The deviation from a linear change noticed when 90 μm Ba-mica was used in the hot pressing is not fully understood as yet. No spinel was detected in the compacts, but higher vaporization of material did occur. A pure Al_2O_3 mixture hot pressed at the same conditions as the composites is also shown on the figure. Apparently the Ba-mica also acts as a sintering aid in this process, allowing for hot pressing at much lower temperatures than would be expected to achieve these densities. Mean $Knoop_{100}$ microhardness values obtained on these same samples are illustrated in Figure 10. They follow essentially the same trends as the density data. Figures 11 and 12 depict four-point flexure strengths and Young's moduli on Ba-mica/Al_2O_3 composites with
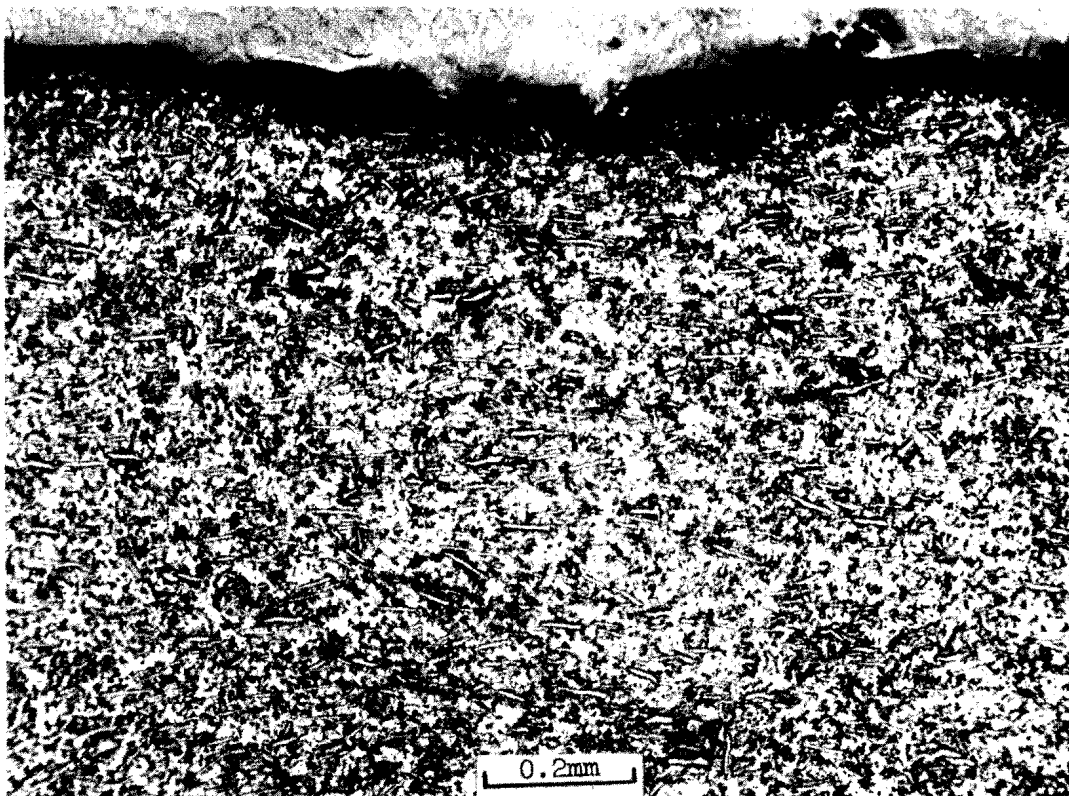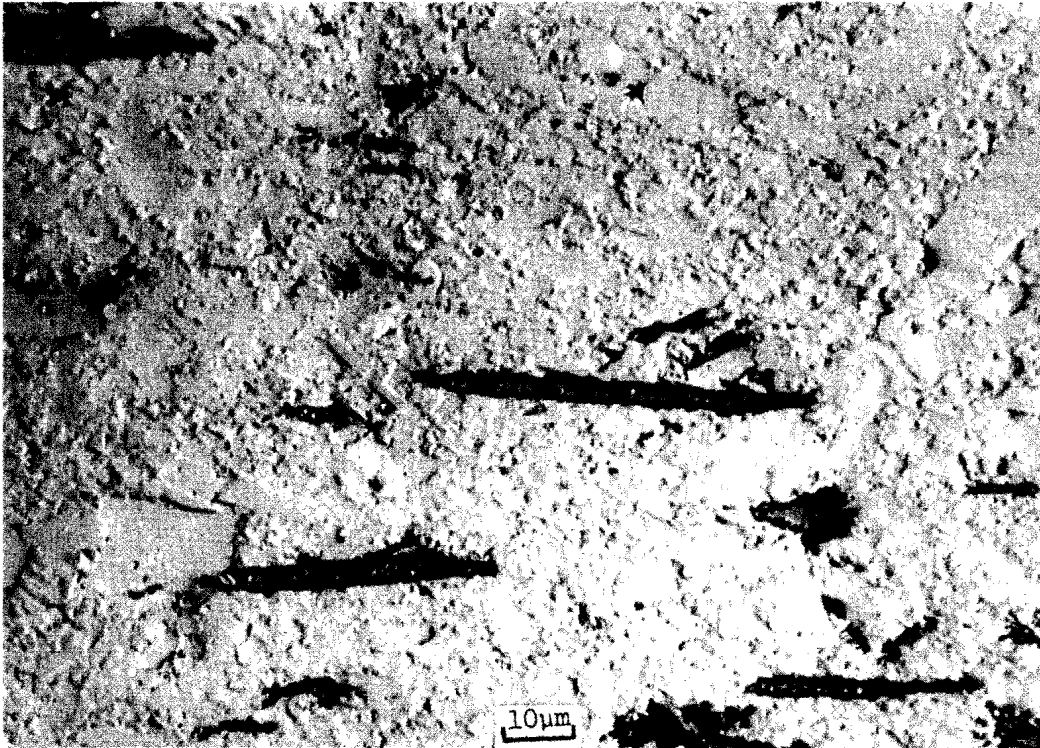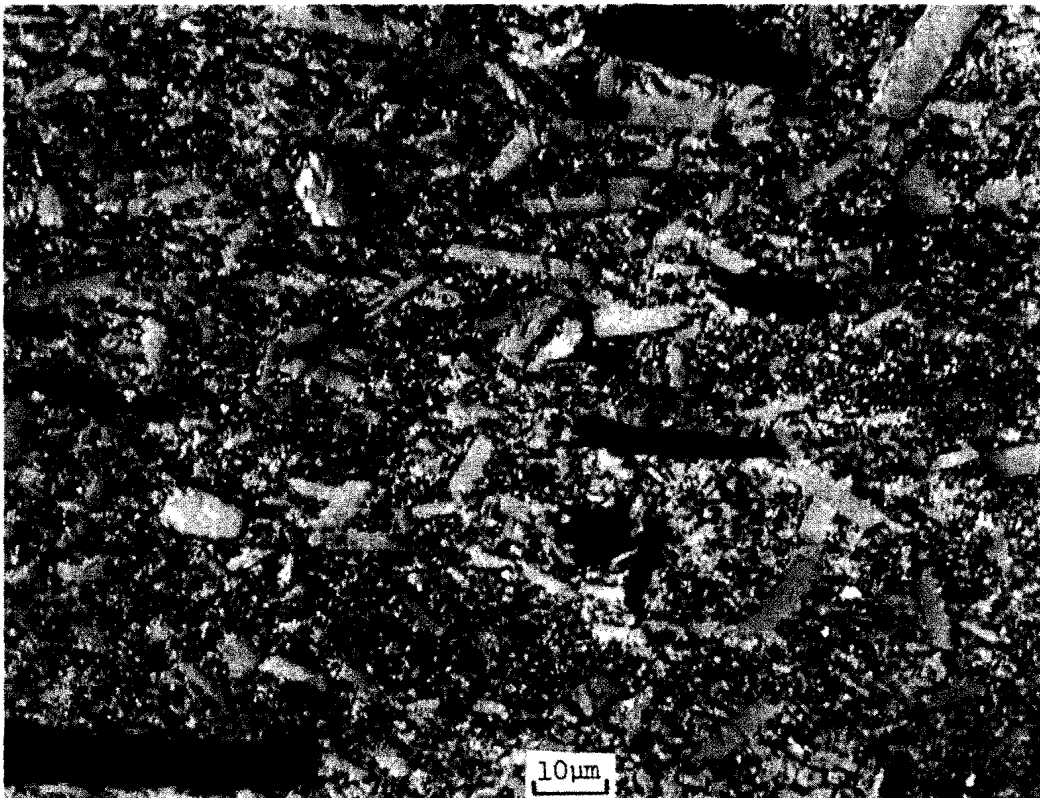

Figure 7. Photomicrograph of Ba-Mica/Al_2O_3 composites with 40 vol % 30 μm Ba-Mica. Hot pressed at 1250 C and 7000 psi.

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a. Multiple relief polishing

b. Etched in boiling phosphoric acid

Figure 8. Photomicrographs of Ba-Mica/Al$_2$O$_3$ composites with 5 vol % 30 μm Ba-Mica Hot pressed at 1250 C and 7000 psi.
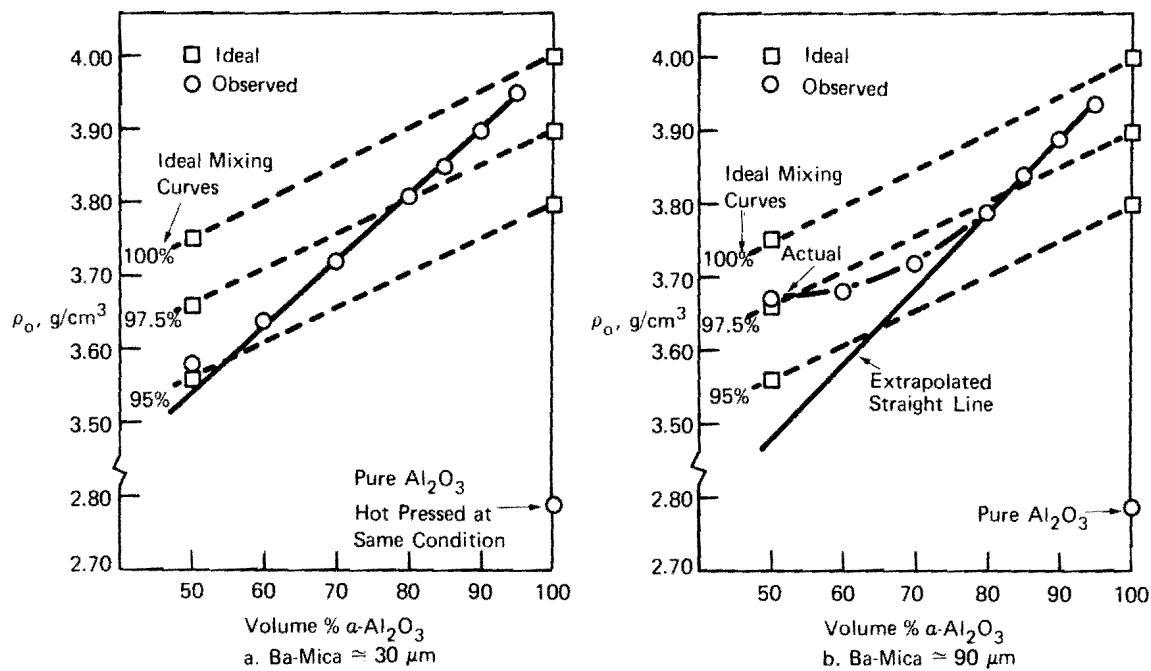
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Figure 9. Observed densities of Ba-Mica/Al_2O_3 composites hot pressed at 1250 C and 7000 psi

30 μm Ba-mica. The flexure strengths and moduli were measured statically on six rectangular bars (1" × 1/8" × 1/8") of each mixture. A dynamic measurement using a sonic resonance technique carried out by Seaton* confirmed the static data. All the samples were prepared by a diamond cut-off wheel, with no further treatment. The only exceptions are the 90 volume percent Al_2O_3 samples which were cut and polished. This different treatment appears to have beneficially affected the data, yielding higher average values of both strength and stiffness. Moduli of 71 and 80 × 10^6 psi were measured on two of these bars.

These last figures clearly show that the properties of Ba-mica/Al_2O_3 can be controlled and tailored to meet specific needs of hardness, strength, and stiffness. One further interesting observation is that the machinability of Al_2O_3 can be improved, while maintaining high strength and stiffness, by the incorporation of Ba-mica. Future mechanical measurements on these materials will test their thermal shock resistance and fracture energy.

The fracture surfaces of broken specimens were also examined by scanning electron microscopy; Figures 13a and b are photographs of a Ba-mica/Al_2O_3 composite with 30 volume percent of 30 μm Ba-mica. These photographs clearly show that the mica flakes are well bonded to the Al_2O_3 matrix without the appearance of any reaction product at the interface. Figure 13b shows several jagged edges on the mica flakes indicating that the mica was not simply pulled out of the Al_2O_3 matrix, but was also fractured as well. This should increase the fracture energy of the material, thereby enhancing the impact strength.

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*Personal communication

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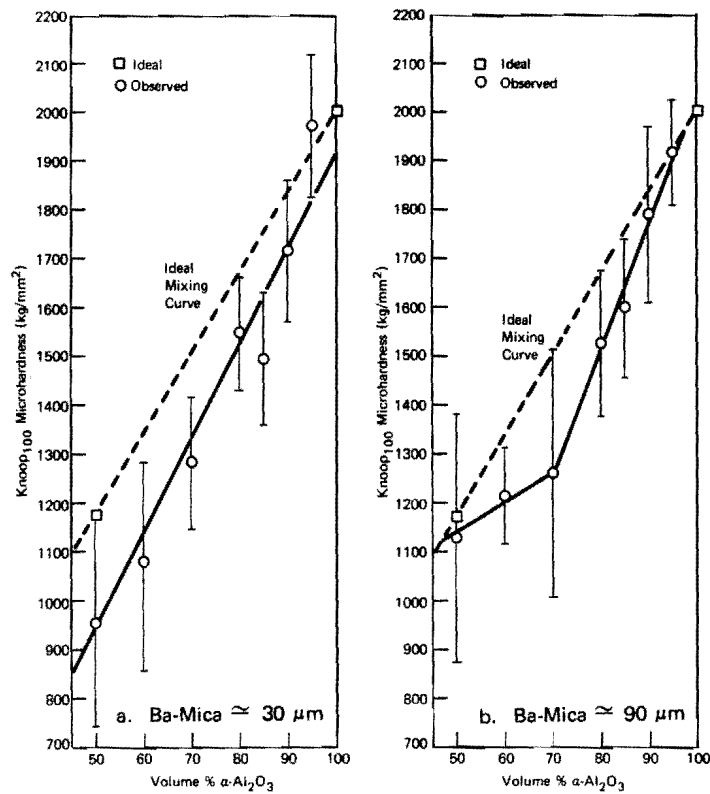
Figure 10. Mean $Knoop_{100}$ microhardness values of $Ba\text{-}Mica/Al_2O_3$ composites hot pressed at 1250 C and 7000 psi
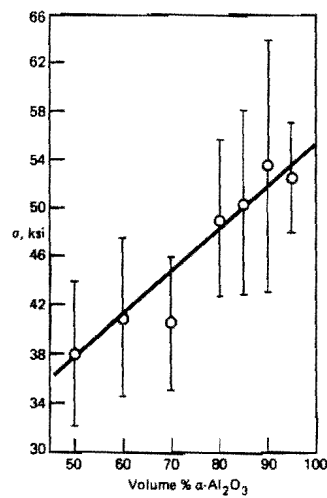
Figure 11. Four-point flexure strengths of Ba-Mica/ Al_2O_3 composites. Ba-Mica $\simeq$ 30 μm
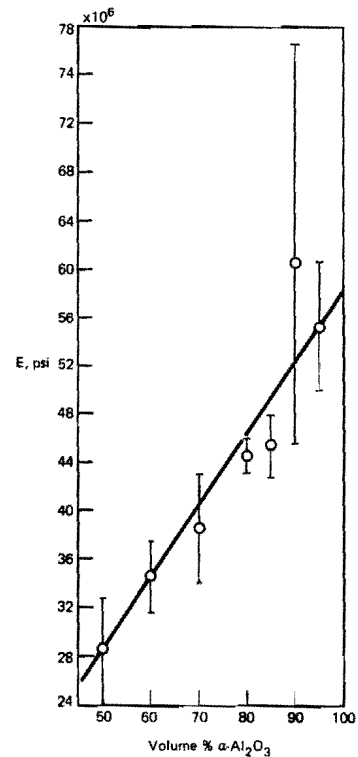
Figure 12. Young's moduli of $Ba\text{-}Mica/Al_2O_3$ composites. Ba-Mica $\simeq$ 30 μm

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a. Bonding at interface
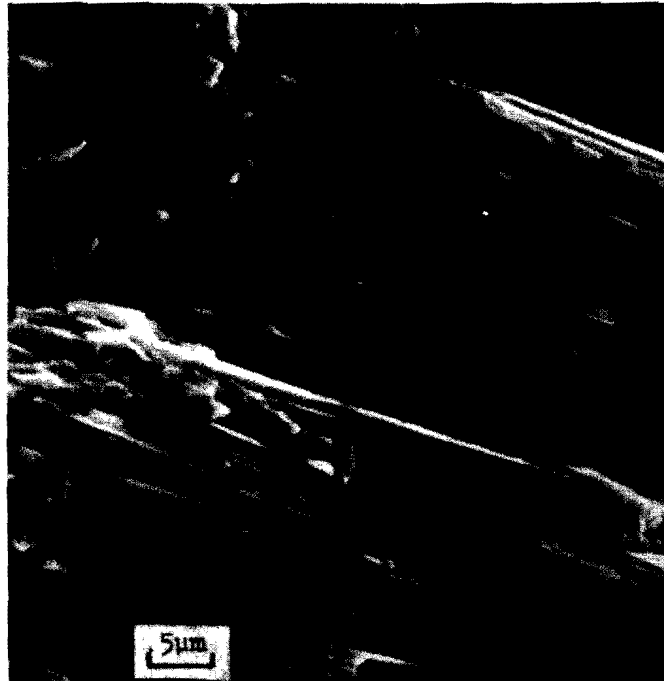
b. Fracture in the mica flakes

Figure 13. SEM fracture surface of Ba-Mica composite with 30 vol % of 30 μm Ba-Mica
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V. CONCLUSIONS

The feasibility of fabricating lamellar ceramic-based composites with synthetic fluorine mica has been demonstrated. Ba-mica/Al_2O_3 composites with greater than 99.5% of theoretical density have been produced with the following ranges of properties: $Knoop_{100}$ hardness from 950 to 1970 kg/mm^2, four-point flexure strength from 38,000 to 53,500 psi, and Young's modulus from 28.6 to 60.5 $\times 10^6$ psi. If the thermal shock resistance of these materials is also enhanced, potential applications include structural components in turbine engines and high temperature bearings.

Reactive hot pressing of K-mica with γ-Al_2O_3 showed that fully dense K-mica/ Al_2O_3 composites cannot be fabricated by hot pressing up to 7000 psi. The work did demonstrate, however, that this technique allows for the fabrication of eutectic-like $MgAl_2O_4$/Al_2O_3 composites 700 C below the eutectic temperature.

VI. ACKNOWLEDGMENTS

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13. ABSTRACT

The feasibility of fabricating lamellar ceramic-based composites with controllable and easily modified properties by reactive hot pressing of gamma-Al$_2$O$_3$ with Ba-mica (BaMg$_3$Al$_2$Si$_2$O$_{10}$F$_2$) has been successfully demonstrated. Cylindrical disks 2 inches in diameter by 1/4-inch thick of Ba-mica/Al$_2$O$_3$ composite material with greater than 99.5% of theoretical density have been produced with the following range of properties: Knoop$_{100}$ hardness from 950 to 1970 kg/mm^2, four-point flexure strength from 38,000 to 53,500 psi, and Young's modulus from 28.6 to 60.5 × 10^6 psi. (Author)

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Flake composites						
Fabrication						
Mica						
Aluminum oxide						
Hot pressing						